

tungsten carbide powder produced by the process, which has an average particle size of 0.8 μm or less and is free of a coarse power having a particle size of more than 1 μm , and which also contains less metal impurities and contains oxygen and nitrogen in a predetermined amount.

REMARKS

The above amendments to the specification, claims, and abstract have been made to correct grammatical errors as amended and to put the application in better condition for examination. No new matter has been added.

Marked-up version of claims under 37 CFR § 1.121(c)(1)(ii) is appended hereto.

In the event that any fees are due in connection with this paper, please charge our Deposit Account No. 18-0013

Respectfully submitted,

Date: April 24, 2002



David K. Benson
Reg. No. 42,314

Rader, Fishman & Grauer PLLC

Lion Building, Suite 501
1233 20th Street, N.W.
Washington, D.C. 20036
Telephone: (202) 955-3750
Facsimile: (202) 955-3751
Customer No. 23353

DKB/nb

APPENDIX

VERSION WITH MARKINGS TO SHOW CHANGES MADE

IN THE SPECIFICATION

The following amendments have been made:

1. Please amend first paragraph on page 2, lines 8 to 12, as follows:

To develop a cemented carbide having a higher strength from the above points of view, the present inventors have focused research on a fine tungsten carbide powder [using] used as a raw powder of a cemented carbide and have obtained the research results shown in the following (a) to (d).

2. Please amend second paragraph bridging pages 3 and 4, page 3, line 21 through page 4, line 12, as follows:

(c) According to a process for producing a fine tungsten carbide powder, which comprises mixing an aqueous solution of ammonium tungstate as a starting material with a carbon powder to form a slurry, drying the slurry to form a precursor mixed with the carbon powder, heating the mixed precursor in [an inert] a non-oxidizing gas atmosphere, thereby causing the reduction and carburization by means of the carbon powder in the mixed precursor to produce a [reduction and carburization] reduced and carburized product composed mainly of tungsten carbide, and finally mixing the [reduction and carburization product with the same] reduced and carburized product with the similar carbon powder used in the preparation of the slurry in a proportion so that W:C is substantially 1:1, and subjecting the mixture to a carburization in a hydrogen atmosphere, it becomes possible to form a high-purity, fine and high-

performance tungsten carbide powder which contains less metal impurities and less coarse WC particles, and which also contains nitrogen and oxygen in trace amounts.

3. Please amend third paragraph on page 5, lines 17 to 23, as follows:

(c) subjecting the precursor to a reduction and carburization [for] by heating to a temperature, at which a reduction and carburization proceed (preferably within a range of 900-1600°C, and more preferably within a range of 1000-1200°C), in a non-oxidizing gas atmosphere (preferably in a mixed gas of a nitrogen gas at normal pressure and a CO gas produced by the reaction) to form a [reduction and carburization] reduced and carburized product, which is substantially free of oxides,

4. Please amend fourth paragraph bridging on pages 5 and 6, page 5, line 24 through page 6, line 4, as follows:

(d) mixing the [reduction and carburization] reduced and carburized product with a carbon powder (preferably carbon black powder) preferably having a purity of at least 99.9% by weight, and more preferably at least 99.99% by weight, in a proportion required to carburize a W_2C component and/or a W component in the [reduction and carburization] reduced and carburized product into WC, and

5. Please amend first paragraph on page 6, lines 5 to 12, as follows:

(e) subjecting the [reduction and carburization] reduced and carburized product mixed with the carbon powder to a carburization for heating to a temperature, at which a carburization proceeds (preferably within a range of 900-1600°C, and more preferably within a range of 1000-1400°C), in a hydrogen atmosphere, thus producing

a fine tungsten carbide powder having an average particle size of 0.8 μm or less, and to a high-performance fine tungsten carbide powder produced by the process.

6. Please amend first paragraph on page 8, lines 1 to 14, as follows:

(c) Content of carbon powder in slurry

When the atomic ratio of carbon to tungsten in ammonium tungstate (C/W) is less than 3, oxides remain in the [reduction and carburization] reduced and carburized product. When oxides exist in the [reduction and carburization] reduced and carburized product, the oxide reacts with hydrogen in the atmosphere in the following step of carburizing with heating to form H_2O , which promotes grain growth of the tungsten carbide powder. Therefore, the average particle size increases to produce WC particles wherein grain growth locally occurs. On the other hand, when the content exceeds 4, the content of free carbon in the [reduction and carburization] reduced and carburized product increases. Therefore, the content is preferably within a range of 3-4.

7. Please amend second paragraph on page 8, lines 16 to 22, as follows:

(d) Drying temperature

The slurry is dried by a simple heating process in air, or by a spray-dry process. When the heating temperature exceeds 350°C , tungsten oxide produced by the decomposition of ammonium tungstate causes grain growth, thus making it difficult to form a fine [reduction and carburization] reduced and carburized product. Therefore, the heating temperature is preferably 350°C or less.

8. Please amend second paragraph bridging on pages 9 and 10, page 9, line 22 through page 10, line 7, as follows:

(g) Maximum size of WC particles

Even if the cemented carbide is produced by using a fine tungsten carbide powder having an average particle size of 0.8 μm or less as a raw material, coarse WC particles included in the cemented carbide [acts] act as the origins of fractures, thereby causing reduction in strength. In the desired fine alloy of the present invention, the maximum particle diameter of WC particles is preferably controlled to 1 μm . As the average particle size of [the fine powder, a value converted from the specific] powders, the Fischer Subsieve Sizer (FSSS) process is familiar. However, for the fine powder, a value converted from the specific surface area in accordance with the BET process or a value measured by SEM is preferably used.

9. Please amend second paragraph on page 12, lines 7 to 12, as follows:

The qualitative analysis of the [reduction and carburization] reduced and carburized products formed by the [reduction and carburization] reduced and carburized was conducted by X-ray diffraction. As a result, it has been confirmed [from the resulting composition formula] that all [reduction and carburization] reduced and carburized products are mainly composed of WC and are substantially free of oxides.

10. Please amend third paragraph on pages 12 and 13, page 12, line 13 through page 13, line 1, as follows:

Subsequently, a CB powder which is the same as that added to the above aqueous solutions of ammonium tungstates was add to the above [reduction and carburization] reduced and carburized products in the proportions shown in Table 1 (which are proportions required to substantially carburize W_2C and W in the [reduction and carburization] reduced and carburized products into WC in the composition formula and denotes a proportion of the content to the total amount of the [reduction and carburization] reduced and carburized products). After mixing using a stirrer, the mixture was subjected to a carburization using the same fixed bed furnace (a horizontal type rotary furnace may be used) in a hydrogen gas atmosphere under 1 atmosphere pressure under the conditions of a predetermined temperature within a range of 900-1600°C for 0.5-1 hours, thereby carrying out the processes 1 to 15 of the present invention.

11. Please amend first paragraph on page 13, lines 1 to 8, as follows:

With respect to the [carburization] carburized products obtained by the processes 1 to 15 of the present invention, X-ray diffraction was conducted. As a result, only diffraction lines of WC were observed. Using six diffraction lines of (001), (100), (110), (111), (211) and (300) among these diffraction lines, lattice constants of an a-axis and a c-axis were determined.

12. Please amend Table 1, the second column from the right, as follows:

Table 1

Class	Formulation of slurry(% by weight)							Reduction and carburization temperature (°C)	Ratio of CB powder to [reduction and carburization] <u>reduced</u> and <u>carburized</u> product (% by weight)	Carburization temperature (°C)
	AMT		APT		CB powder					
	Purity (% by weight)	Concentration of aqueous solution (% by weight)	Purity (% by weight)	Concentration of aqueous solution (% by weight)	Purity (% by weight)	C/W ratio				
Process of the present invention	1	99.915	35	—	—	99.913	3.9	900	0.09	1200
	2	99.952	35	—	—	99.955	3.5	1000	0.23	1200
	3	99.977	35	—	—	99.972	3.3	1300	0.42	1600
	4	—	—	99.911	20	99.915	3.7	1100	0.14	1100
	5	—	—	99.956	20	99.954	3.5	1100	0.21	1300
	6	—	—	99.975	20	99.975	3.2	1100	0.37	1200
	7	99.995	20	—	—	99.993	4.0	900	0.05	900
	8	99.995	35	—	—	99.993	3.3	1000	0.17	1100
	9	99.995	50	—	—	99.993	3.6	1100	0.28	1000
	10	99.995	60	—	—	99.993	3.2	1300	0.43	1300
	11	99.995	70	—	—	99.993	3.0	1400	0.48	1400
	12	—	—	99.996	20	99.997	3.2	1000	0.11	1400
	13	—	—	99.996	20	99.997	3.2	1200	0.26	1200
	14	—	—	99.996	20	99.997	3.5	1200	0.32	1300
	15	—	—	99.996	20	99.997	3.3	1600	0.36	950

IN THE CLAIM

Claim 1 has been amended as follows:

1. (Amended) A process for producing a fine tungsten carbide powder, comprising the steps of:

(a) mixing an aqueous ammonium tungstate solution with a carbon powder in a proportion to reduce and carburize ammonium tungstate to form a slurry,

(b) drying the slurry to prepare a precursor,

(c) subjecting the precursor to a [reduction and carburization] reduced and carburized for heating to a temperature, at which a [reduction and carburization] proceeds, in a non-oxidizing gas atmosphere to form a [reduction and carburization] product,

(d) mixing the [reduction and carburization] reduced and carburized product with a carbon powder in a proportion required to carburize a W_2C component and/or a W component in the [reduction and carburization] reduced and carburized product into WC, and

(e) subjecting the [reduction and carburization] reduced and carburized product mixed with the carbon powder to a carburization [for] by heating to a temperature, at which a carburization proceeds, in a hydrogen atmosphere.

IN THE ABSTRACT OF THE DISCLOSURE

A process is provided for producing a fine tungsten carbide powder, which comprises the steps of drying a slurry, which is obtained by mixing an aqueous ammonium tungstate solution with a carbon powder, at low temperature, to form a

precursor, mixing a [reduction and carburization] reduced and carburized product, which is obtained by reducing and oxidizing the precursor in [an inert] a non-oxidizing gas, with a carbon powder in a proportion required to substantially carburize the entire tungsten component into tungsten carbide (WC), and carburizing the mixture; and a high-performance fine tungsten carbide powder produced by the process, which has an average particle size of 0.8 μm or less and is free of a coarse powder having a particle size of more than 1 μm , and which also contains less metal impurities and contains oxygen and nitrogen in a predetermined amount.